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IS 1448-1 (1971): Neutralization Number by Potentiometric Titration - Part I [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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(Reaffirmed 2002)

NEUTRALIZATION NUMBER BY POTENTIOMETRIC TITRATION

[P:1]

(*First Revision*)

First Reprint AUGUST 1982
UDC 665·6/·7 : 543·257·1

Adapted from the Joint ASTM-IP Standard No. ASTM D664/58-IP/177/64

(In order to align the method with the joint ASTM-IP standard the title of this method has been changed.)

1. SCOPE

1.1 This method covers the determination of acidic or basic constituents in petroleum products and lubricants (*see* **1.2.1**). The method resolves these constituents into groups having weak-acid, strong-acid, weak-base, and strong-base ionization properties, provided the dissociation constants of the more strongly acidic or basic compounds are at least 1 000 times that of the next weaker groups.

1.2 The method may be used to indicate relative changes that occur in an oil during use under oxidizing conditions regardless of the colour or other properties of the resulting oil. Although the titration is made under definite equilibrium conditions, the method is not intended to measure an absolute acidic or basic property which may be used to predict performance of an oil under service conditions. No general relationship between bearing corrosion and acid or base number is known.

1.2.1 In new and used oils, the constituents that may be considered to have acidic characteristics include organic and inorganic acids, esters, phenolic compounds, lactones, resins, salts of heavy metals, salts of ammonia and other weak bases, acid salts of polybasic acids, and additives, such as inhibitors and detergents. Similarly, constituents that may be considered to have basic properties include organic and inorganic bases, amino compounds, salts of weak acids (soaps), basic salts of polyacidic bases, salts of heavy metals, and additives, such as inhibitors and detergents.

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2. TERMINOLOGY

2.0 For the purpose of this method, the following definitions shall apply.

2.1 Total Acid Number — The quantity of base, expressed in milligrams of potassium hydroxide, that is required to neutralize all acidic constituents present in one gram of the sample.

2.2 Strong Acid Number — The quantity of base, expressed in milligrams of potassium hydroxide, that is required to neutralize the strong acid constituents present in one gram of the sample.

2.3 Total Base Number — The quantity of acid, expressed in terms of the equivalent number of milligrams of potassium hydroxide, that is required to neutralize all basic constituents present in one gram of the sample.

2.4 Strong Base Number — The quantity of acid, expressed in terms of the equivalent number of milligrams of potassium hydroxide, that is required to neutralize the strong base constituents present in one gram of the sample.

3. OUTLINE OF THE METHOD

3.1 The sample is dissolved in a mixture of toluene and *iso*propyl alcohol containing a small amount of water and titrated potentiometrically with alcoholic potassium hydroxide or hydrochloric acid solution using a glass indicating electrode and a calomel reference electrode. The meter readings are plotted against the respective volumes of titrating solution and the end points are taken at the inflections in the resulting curve. When no definite inflections are obtained, end points are taken at meter readings corresponding to the end points in the titration of standard non-aqueous acidic and basic buffer solutions.

4. APPARATUS

4.1 Cell Assembly — Consisting of glass electrode, calomel electrode, stirrer, burette, beaker and stand. The cell assembly is shown in Fig. 1 and its different parts are described in **4.1.1** to **4.1.6**.

4.1.1 *Glass Electrode* — A pencil type glass electrode (*C* in Fig. 1), 125 to 180 mm in length and 8 to 14 mm in diameter. The body of the electrode shall be made of a chemically resistant glass tube with a wall thickness of 1 to 3 mm. The end dipping into the solution shall be closed with a hemisphere of Corning 015 glass or equivalent sealed on to the electrode tube and the radius of this hemisphere shall be about 7 mm. The thickness of the glass in the hemisphere shall be great enough so that the resistance of the hemisphere is 100 to 1 000 MΩ at 25°C. The electrode shall contain a reproducible, permanently sealed liquid cell for making electrical connection

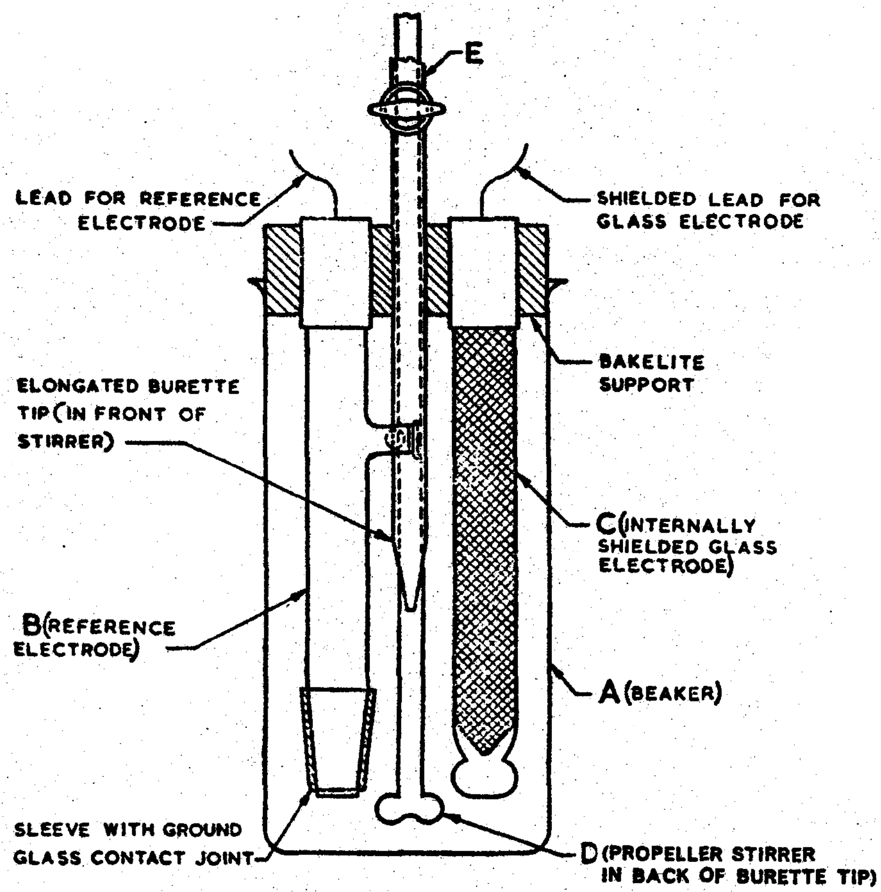
E
LEAD FOR REFERENCE ELECTRODE
SHIELDED LEAD FOR GLASS ELECTRODE
BAKELITE SUPPORT
ELONGATED BURETTE TIP (IN FRONT OF STIRRER)
C (INTERNALLY SHIELDED GLASS ELECTRODE)
B (REFERENCE ELECTRODE)
A (BEAKER)
SLEEVE WITH GROUND GLASS CONTACT JOINT
D (PROPELLER STIRRER IN BACK OF BURETTE TIP)

FIG. 1 CELL FOR POTENTIOMETRIC TITRATION

with the inner surface of the hemisphere. The entire electrical connection from the sealed contact cell to the meter terminal shall be surrounded by an electrical shield that will prevent electrostatic interference when the shield is grounded. The shield shall be insulated from the electrical connection by insulating material of the highest quality, such as rubber and glass, so that the resistance between the shield and the entire length of the electrical connection is greater than 50 000 MΩ.

4.1.2 *Calomel Electrode* — A pencil type calomel electrode (*B* in Fig. 1) 125 to 180 mm in length and 8 to 14 mm in diameter. This electrode shall be made of glass and provided with an external, removable glass sleeve on the sealed end which is dipped into the titration solution. The glass sleeve shall be 8 to 25 mm in length, slightly tapered, and ground to fit the electrode so that the sealed end of the electrode protrudes 2 to 20 mm beyond the sleeve. The ground surface shall be continuous and free from smooth spots. At a point midway between the extremities of the ground surface, the electrode tube shall be pierced by one or more holes each one millimetre in diameter. The electrode shall contain the necessary mercury, calomel, and electrical connection to the mercury, all arranged in a permanent manner. The electrode shall be filled almost to capacity with saturated potassium chloride electrolyte and shall be equipped with a stoppered port through which the electrolyte may be replenished. When suspended in the air and with the sleeve in place, the electrode shall not leak electrolyte at a rate greater than one drop in 10 minutes.

4.1.3 *Stirrer* — A variable speed mechanical stirrer of any suitable type, equipped with a glass, propeller type stirring paddle (*D* in Fig. 1). A propeller with blades 6 mm in radius and set at a pitch of 30 to 45 degrees is satisfactory. If electrical stirring apparatus is used, it shall be electrically correct and grounded so that connecting or disconnecting the power to the motor will not produce a permanent change in meter reading during the course of the titration.

4.1.4 *Burette* — A 10-ml burette (*E* in Fig. 1) graduated in 0·05 ml divisions, and calibrated with an accuracy of ±0·02 ml. The burette shall have a glass stopcock and shall have a tip that extends 100 to 130 mm beyond the stopcock.

4.1.5 *Titration Beaker* — A 250 to 300-ml, tall form, lipless beaker (*A* in Fig. 1) made of chemically resistant glass. The beaker shall be 116 to 120 mm in height and 62 ± 3 mm inside diameter below the flared portion with sides tapering in at an angle of approximately one degree from the vertical.

4.1.6 *Titration Stand* — A suitable stand to support the electrodes, stirrer, and burette in the position shown in Fig. 1. An arrangement that allows the removal of the beaker without disturbing the electrodes, burette, and stirrer is desirable.

4.2 Meter* — A voltmeter or potentiometer that will operate with an accuracy of ±0·005 V and a sensitivity of ±0·002 V, over a range of at least ±0·5 V, when the meter is used with the electrodes specified in **4.1.1** and **4.1.2** and when the resistance between the electrodes falls within the range of 0·2 to 20 MΩ. The meter shall be protected from stray electrostatic fields so that no permanent change in the meter readings over the entire

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*The Beckman *p*H meter (model M or G) manufactured by the National Technical Laboratories, Pasadena, Cal., and the Dual Titrometer manufactured by the Precision Scientific Co, Chicago 47, Ill. or any other suitable *p*H meter may be used.

operating range is produced by touching, with a grounded* lead, any part of the exposed surface of the glass electrode, the glass electrode lead, the titration stand, or the meter. A desirable apparatus may consist of a continuous reading electronic voltmeter with specified range, accuracy, and sensitivity, that is designed to operate on an input of less than 5 × 10^{-12} A when an electrode system having 1 000 MΩ resistance is connected across the meter terminals, which is provided with a metal shield connected to the ground* and with a satisfactory terminal to connect the shielded connection wire from the glass electrode to the meter without interference from the presence of the external electrostatic fields.

5. REAGENTS

5.0 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960†) shall be used.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

5.1 Standard Alcoholic Potassium Hydroxide Solution (0·1 N) — Add 6 g of potassium hydroxide (KOH) to approximately one litre of anhydrous *iso*propyl alcohol. Boil gently for 10 minutes to effect solution. Allow the solution to stand for two days and then filter the supernatant liquid through a fine sintered glass funnel. Store the solution in a chemically resistant glass bottle. Dispense in a manner such that the solution is protected from atmospheric carbon dioxide (CO_2) by means of a guard tube containing sodalime or soda asbestos (Ascarite, Carbosorb, or Indecarb), and such that it does not come into contact with cork, rubber, or saponifiable stopcock grease. Standardize frequently enough to detect normality changes of 0·000 5 by potentiometric titration of weighed quantities of potassium hydrogen phthalate dissolved in carbon dioxide free water.

5.2 Standard Alcoholic Potassium Hydroxide Solution (0·2 N) — Prepare, store, and standardize as directed in **5.1**, but use 12 to 13 g of potassium hydroxide to approximately one litre of *iso*propyl alcohol.

5.3 Standard Alcoholic Hydrochloric Acid Solution (0·1 N) — Mix 9 ml of hydrochloric acid (sp gr 1·19, *see* IS : 265-1962‡) with one litre of anhydrous *iso*propyl alcohol. Standardize frequently enough to detect normality changes of 0·000 5 by potentiometric titration of approximately 8 ml (accurately measured) of the standard 0·1 N alcoholic potassium hydroxide solution diluted with 125 ml of carbon dioxide free water.

5.4 Standard Alcoholic Hydrochloric Acid Solution (0·2 N) — Prepare and standardize as directed in **5.3**, but use 18 ml of hydrochloric acid (sp gr 1·19, *see* IS : 265-1962‡).

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*'Grounded' or 'connected to the ground' means connected through a resistance of not more than 100 ohms to a standard ground potential such as that of a water-service pipe.

†Specification for water, distilled quality (*revised*).

‡Specification for hydrochloric acid (*revised*).

5.5 Titration Solvent — Add 500 ml of toluene and 5 ml of water to 495 ml of anhydrous *iso*propyl alcohol. The titration solvent should be made up in large quantities, and its blank value determined daily by titration prior to use.

5.5.1 A titration solvent in which chloroform is used in place of toluene may be required to completely dissolve certain heavy residues or asphaltic materials.

5.6 Buffer Stock Solution *A* — Accurately weigh 24·2 ± 0·1 g of 2, 4, 6-trimethylpyridine (γ-collidine), and transfer to a one-litre volumetric flask containing 100 ml of *iso*propyl alcohol. Using a one-litre graduated cylinder, add to the flask, while continuously stirring its contents, $150/N_1 \pm 5$ ml of 0·2 N standard alcoholic hydrochloric acid solution (N_1 being the exact normality of hydrochloric acid found by standardization). Dilute to the mark with *iso*propyl alcohol, and mix thoroughly.

5.7 Buffer Stock Solution *B* — Accurately weigh 27·8 ± 0·1 g of *m*-nitro-phenol and transfer to a one-litre volumetric flask containing 100 ml of *iso*propyl alcohol. Using a 250-ml graduated cylinder, add to the flask while continuously stirring its contents $50/N_2 \pm 1$ ml of 0·2 N standard alcoholic potassium hydroxide solution (N_2 being the exact normality of potassium hydroxide solution found by standardization). Dilute to the mark with *iso*propyl alcohol, and mix thoroughly.

5.8 Non-aqueous Acidic Buffer Solution — Add 10 ml of buffer stock solution *A* to 100 ml of titration solvent. Use within one hour.

5.9 Non-aqueous Basic Buffer Solution — Add 10 ml of buffer stock solution *B* to 100 ml of titration solvent. Use within one hour.

5.10 Potassium Chloride Electrolyte — Prepare a saturated solution of potassium chloride (KCl) in water.

5.11 2, 4, 6-Trimethylpyridine (γ-Collidine) (mol wt 121·18) — conforming to the following requirements:

Boiling range	168 — 170°C
Refractive index N_D^{20}	1·498 2 ± 0·000 5
Colour	Colourless

Note — Store the reagent over activated alumina and keep in a brown glass bottle.

5.12 *m*-Nitrophenol (mol wt 139·11) — conforming to the following requirements:

Melting point	96 — 97°C
Colour	Pale yellow

Note — Store the reagent in brown glass bottle.

6. PREPARATION OF ELECTRODE SYSTEM

6.1 Maintenance of Electrodes — The glass electrode shall be cleaned (*see* **6.1.1**) at frequent intervals (not less than once every week during continual use) by immersing in cold chromic acid cleaning solution. The calomel electrode should be drained at least once every week and refilled with fresh potassium chloride (KCl) electrolyte. The electrolyte level in the calomel electrode shall be kept above that of the liquid in the titration beaker at all times. When not in use, the lower halves of the electrodes shall be immersed in water and not allowed to remain immersed in titration solvent for any appreciable period of time between titrations. Although the electrodes are not extremely fragile, they should be handled carefully at all times.

6.1.1 Cleaning the electrodes thoroughly, keeping the ground glass joint free of foreign materials, and regular testing of the electrodes are very important in obtaining reproducible potentials, since contamination may introduce uncertain, erratic and unnoticeable liquid contact potentials. While this is of secondary importance when end points are chosen from inflection points in the titration curve, it may be quite serious when end points are chosen at arbitrarily fixed cell potentials.

6.2 Preparation of Electrodes — Before and after using, wipe the glass electrode thoroughly with a clean cloth, or a soft absorbent tissue, and rinse with water. Wipe the calomel reference electrode with a cloth or tissue, carefully remove the ground glass sleeve, and thoroughly wipe both ground surfaces. Replace the sleeve loosely and allow a few drops of electrolyte to drain through to flush the ground glass joints (*see* **6.1.1**). Wet the ground surfaces thoroughly with electrolyte, set the sleeve firmly in place, and rinse the electrode with water. Prior to each titration, soak the prepared electrode in water for at least five minutes immediately before use, and touch the tips of the electrodes with a dry cloth or tissue to remove the excess water.

6.3 Testing of Electrodes — The meter-electrode combination shall be tested (*see* **6.1.1**) when first put into use, or when new electrodes are installed, and retested at intervals thereafter by dipping the electrodes into a well-stirred mixture of 100 ml of the titration solvent and 1·0 to 1·5 ml of 0·1 N standard alcoholic potassium hydroxide solution. If the meter-electrode combination is suitable for use, the potential between the electrodes will have changed by more than 0·480 V from the potential between the same electrodes when dipped in the non-aqueous acidic buffer solution (*see* Note).

NOTE — Considerably more sensitive electrodes are now available that will show a potential change of at least 0·590 V under these conditions, and their use is recommended.

7. STANDARDIZATION OF APPARATUS

7.1 Determination of Meter Readings for the Non-aqueous Buffer Solutions Corresponding to Acid and Base End Points — To ensure comparable selection of end points when definite inflection points are not obtained in the titration curve, determine daily, for each electrode pair, the meter readings obtained with the non-aqueous acidic and basic buffer solutions (*see* **7.1.1**). Prepare the electrodes as described in **6.2**, immerse them in the non-aqueous buffer solution, and stir for five minutes, maintaining the temperature within 2°C of that at which the titrations are to be made. Read the cell voltage. The readings so obtained are taken as the end points in titration curves having no inflection points.

7.1.1 The response of different glass electrodes to hydrogen ion activity is not the same. Therefore, it is necessary to establish regularly for each electrode system the meter readings corresponding to the buffer solutions arbitrarily selected to represent acidic or basic end points.

8. PREPARATION OF SAMPLE OF USED OIL

8.1 Strict observance of the sampling procedure is necessary, since the sediment itself is acidic or basic or has adsorbed acidic or basic material from the sample. Failure to obtain a representative sample causes serious errors.

8.1.1 As used oil may change appreciably in storage, samples should be tested as soon as possible after removal from the lubricating system; and the dates of sampling and testing should be noted.

8.2 Heat the sample (*see* **8.2.1**) of used oil to 60 ± 5°C in the original container until all of the sediment is homogeneously suspended in the oil. If the original container is a can, or if it is glass and more than three-fourths full, transfer the entire sample to a clear glass bottle having a capacity at least one-third greater than the volume of the sample. Transfer all traces of sediment from the original container to the bottle by violent agitation of portions of the sample in the original container.

8.2.1 When samples are visibly free from sediment, the heating procedure described may be omitted.

8.3 After complete suspension of all sediment, strain the sample or a convenient aliquot through IS Sieve 15 (aperture 151 microns) for the removal of large contaminating particles.

9. PROCEDURE FOR TOTAL ACID NUMBER AND STRONG ACID NUMBER

9.1 Into the titration beaker, introduce a weighed quantity of sample as prescribed below and add 125 ml of titration solvent (*see* **5.5.1**). Prepare the electrodes as directed in **6.2**. Place the beaker on the titration stand and adjust its position so that the electrodes are about half immersed. Start the stirrer, and stir throughout the determination at a rate sufficient to produce vigorous agitation without spattering and without stirring air into the solution:

Acid Number or Base Number	*Size of Sample*	*Sensitivity of Weighing*
	g	g
0·05 to 1·0	20·0 ± 2·0	0·10
1·0 „ 5·0	5·0 ± 0·5	0·02
5 „ 20	1·0 ± 0·1	0·005
20 „ 100	0·25 ± 0·02	0·001
100 „ 250	0·10 ± 0·01	0·000 5

9.2 Fill the burette with standard alcoholic potassium hydroxide solution (0·1 N) and place the burette in position in the titration assembly, taking care that the tip is immersed about 25 mm in the liquid in the beaker. Record the initial burette and meter (cell potential) readings. Add suitable small portions of standard alcoholic potassium hydroxide solution (0·1 N) and after waiting until a constant potential has been established (*see* **9.2.1**), record the burette and meter readings. At the start of the titration and in any subsequent regions (inflections) where 0·1 ml of standard alcoholic potassium hydroxide solution (0·1 N) consistently produces a total change of more than 0·03 V (corresponding to 0·5 *p*H scale units) in the cell potential, add 0·05 ml portions. In the intermediate regions (plateaus) where 0·1 ml changes the cell potential less than 0·03 V, add larger portions sufficient to produce a total potential change approximately equal to, but not greater than 0·03 V. Titrate in this manner until the potential changes less than 0·005 V (corresponding to 0·1 *p*H scale units) per 0·1 ml, and the cell potential indicates that the solution is more basic than the non-aqueous basic buffer solution. Remove the titrated solution, rinse the electrodes with *iso*propyl alcohol, and immerse the electrodes in water.

9.2.1 Consider the cell potential as constant when it changes less than 0·005 V (corresponding to 0·1 *p*H scale units) per minute. This may require approximately 1 to 2 minutes per 0·1 V change in potential (corresponding to 1·7 *p*H scale units) when adding 0·05 ml increments; one millilitre increments may require 3 to 5 minutes.

9.3 Blank — Make a blank titration of 125 ml of titration solvent, adding standard alcoholic potassium hydroxide solution (0·1 N) in 0·05 ml increments and recording the meter and burette readings when the former becomes constant after each increment.

9.3.1 An oil may have both a weak-acid number and a weak-base number if the initial meter reading falls between the readings of the acidic and basic non-aqueous buffer solutions. If the initial meter reading indicates that the solution is more basic than the basic buffer solution or more acidic than the acidic buffer solution, the oil may contain mixtures of strong and weak bases or strong and weak acids, respectively.

10. PROCEDURE FOR TOTAL BASE NUMBER AND STRONG BASE NUMBER

10.1 Proceed as directed in **9**, but titrate with standard alcoholic hydrochloric acid solution (0·1 N), adding it at the same rate as specified for standard alcoholic potassium hydroxide solution.

10.2 Blank — Make a blank titration of 125 ml of titration solvent, adding standard alcoholic hydrochloric acid solution (0·1 N) in 0·05 ml increments in a manner comparable to that specified in **9.3**.

11. CALCULATIONS

11.1 Plot the volumes of the acid or base titrating solution, or both (*see* **9.3.1**), added against the corresponding meter readings (*see* Fig. 2). Mark as an end point any inflection point (*see* **11.1.1**) on the titration curve which occurs near the cell voltages representing the non-aqueous acidic and basic buffer solutions (*see* **7.1**). If no inflection appears (such as in the case of Curve B in Fig. 2), mark end points at these meter readings corresponding to the two non-aqueous buffer solutions.

11.1.1 An inflection point is generally recognizable by inspection whenever several successive 0·05 ml increments each produces a cell potential change greater than 0·015 V (corresponding to 0·25 *p*H scale units) and at least 30 percent greater than those produced by previous or subsequent increments of the same size. Generally, definite inflection points may be discerned only in regions where in crements of the same size are used.

11.2 Calculate the total acid number and strong acid number as follows:

Total acid number, milligrams of potassium hydroxide per gram of sample $= \dfrac{56 \cdot 1 \times (A - B) \times N}{W}$

Strong acid number, milligrams of potassium hydroxide per gram of sample $= \dfrac{56 \cdot 1 \times (CN + Dn)}{W}$

where

A = volume in millilitres of standard alcoholic potassium hydroxide solution used to titrate the sample to an end point that occurs at the meter reading corresponding to the basic non-aqueous buffer solution,

B = volume in millilitres corresponding to A for the blank titration,

N = normality of standard alcoholic potassium hydroxide solution,

W = weight in grams of the sample,

C = volume in millilitres of standard alcoholic potassium hydroxide solution used to titrate the sample to an end point that occurs at a meter reading corresponding to the acidic non-aqueous buffer solution,

D = volume in millilitres of alcoholic standard hydrochloric acid solution used to titrate the solvent blank to the end point corresponding to C, and

n = normality of standard alcoholic hydrochloric acid solution.

11.3 Calculate the total base number and strong base number as follows:

$$\text{Total base number, milligrams of potassium hydroxide per gram of sample} = \frac{56{\cdot}1 \times (E - F) \times N}{W}$$

$$\text{Strong base number, milligrams of potassium hydroxide per gram of sample} = \frac{56{\cdot}1 \times (GN + Hn)}{W}$$

where

E = volume in millilitres of standard alcoholic hydrochloric acid solution used to titrate the sample to an end point which occurs at a meter reading corresponding to the acidic non-aqueous buffer solution,

F = volume in millilitres corresponding to E for the blank titration,

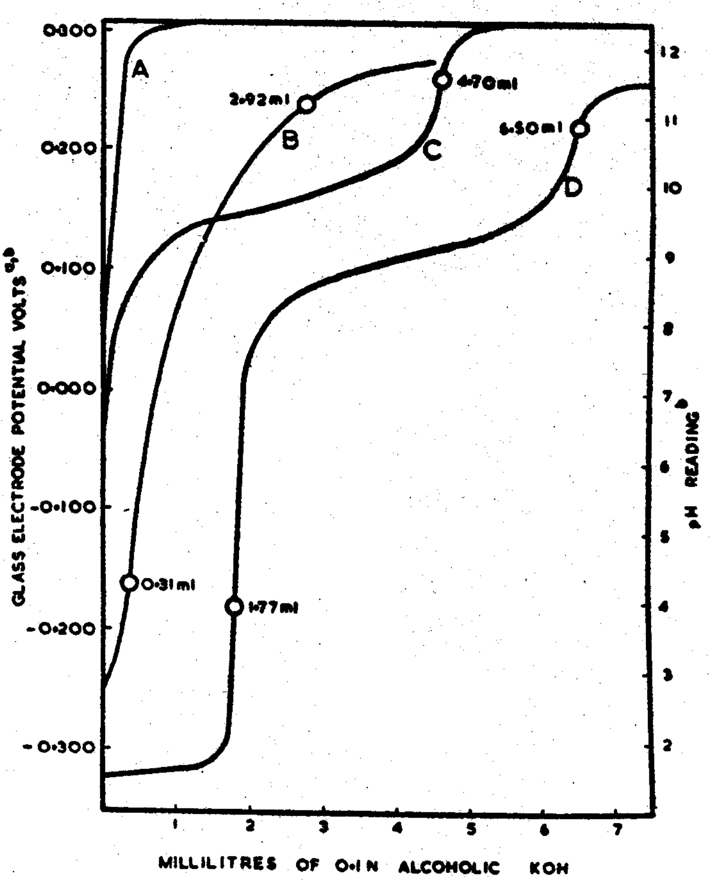
N = normality of standard alcoholic hydrochloric acid solution,

W = weight in grams of the sample,

G = volume in millilitres of alcoholic hydrochloric acid used to titrate the sample to an end point which occurs at a meter reading corresponding to the basic non-aqueous buffer solution,

H = volume in millilitres of alcoholic standard potassium hydroxide solution used to titrate the solvent blank to the end point corresponding to G, and

n = normality of standard alcoholic potassium hydroxide solution.



A — Blank on 125 ml of titration solvent.

B — 10·00 g of used crankcase oil plus 125 ml of titration solvent. Since no sharp inflections are apparent the end points are chosen at the meter readings which were obtained with the two non-aqueous buffers.

C — 10·00 g of oil containing a weak acid plus 125 ml of titration solvent. The end point is chosen at the most vertical portion of the inflection.

D — 10·00 g of oil containing weak and strong acids plus 125 ml of titration solvent. The end points are chosen at the most vertical portions of the two inflections.

a — On some meters, the voltage sign is reversed.

b — In some instruments, the relation between glass electrode potential and *p*H reading is only approximately as shown.

Fig. 2 Illustrative Titration Curve

12. REPORT

12.1 Report results as 'total acid number', 'strong acid number', 'total base number', 'strong base number' IS : 1448 [P : 1].

13. PRECISION

13.1 Good Inflection in Titration Curve — When good inflections are obtained in the titration curves, results shall not differ from the mean by more than the following amounts:

Acid or Base Number	*Repeatability*	*Reproducibility*
0·05 to 1·0	0·02	0·04
1·0 „ 5·0	0·1	0·2
5 „ 20	0·5	1
20 „ 100	2	4
100 „ 250	5	10

13.2 Poor Inflection in Titration Curve — When poor inflections are obtained in the titration curves, the values for repeatability and reproducibility will be larger than those given in **13.1**.

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